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In re Serial Number.

10/655,988

Application to Reissue U.S. Pat. 5,405,911

Filed: Sept. 4, 2003

Inventor: Handlin, Jr., et al.

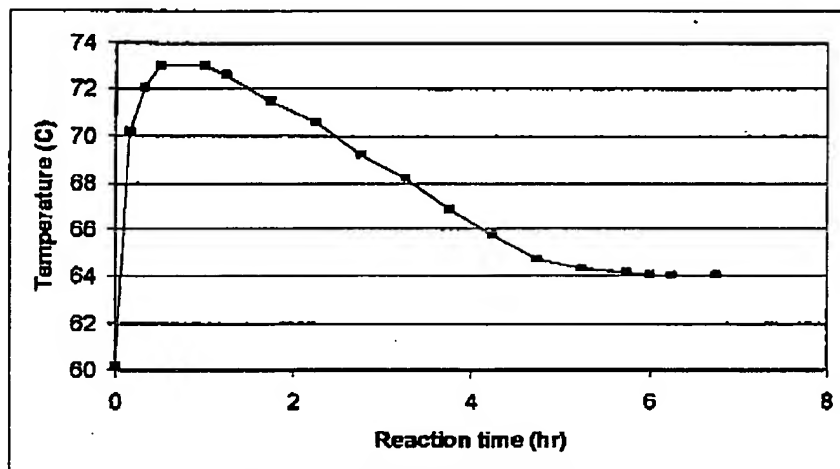
For: Butadiene Polymers Having Terminal Functional Groups

SUPPLEMENTAL YOO DECLARATION IN SUPPORT OF PROTEST

I, Taejun Yoo, hereby declare and say that:

1. I am skilled in the art of polymerization of butadiene and testing of properties of butadiene polymers.
2. I was awarded a Ph.D. in Polymer Sciences by the University of Akron in 1994, my doctoral thesis relating to anionic polymerization of polybutadienes.
3. I did post-doctoral research at Rensselaer Polytechnic Institute,.
4. Following my post-doctoral research at Rensselaer, I was employed in South Korea.
5. Following my employment in South Korea, I did additional post-doctoral research at University of Akron.
6. Since 2001, I have been employed by Sartomer Company in its research department conducting experimental work in the field of polybutadienes.
7. I am familiar with a publication entitled "The Hydrogenation of OH-Terminated Telechelic Polybutadienes in the Presence of a Homogenous Hydrogenation Catalysts Based on Tris(triphenylphosphine)rhodium Chloride" by Karel Bouchal, Michal Ilavsky and Eva Zurková. Die Angewandte Makromolekulare 165, 165-180 (1989), which, I have been told by our attorney, was cited by reissue-applicants in a Supplemental Information Disclosure Statement as reference AM. Bouchal, et al. disclose on page 167 the synthesis of hydroxyl terminated telechelic polybutadienes prepared by anionic polymerization of butadiene on dilithium catalyst and functionalized with ethylene oxide having $M_n=5100$, functionality distribution of $f_0=1$ mol-%, $f_1=9$ mol-%, and $f_2=90$ mol-% (i.e., "about two or more terminal functional groups per molecule"), with 1,2-addition of 59.1%. I obtained such polymer and hydrogenated it to a degree of hydrogenation of greater than 98%.

8. The hydrogenation I employed differed from that of Bouchal, et al, only in the fact that I used a Ru/Carbon hydrogenation catalyst.
9. The details of the hydrogenation I employed were
 - a. A 10% heptane solution of hydroxyl-terminated polybutadiene Krasol LBH-P 2000 or LBH-P 5000 was reacted at a temperature of 70°C, hydrogen pressure of 270 psi, using as catalyst 1.5 g of 5% Ru/Carbon. A 10% heptane solution of Krasol LBH-P2000 or 5000 (30g of the hydroxyl-terminated polybutadiene) and catalyst were added into a pressure vessel.
 - b. The vessel temperature was increased to 70°C with stirring in the presence of nitrogen. The nitrogen purging process was repeated three times during the process of heating. After the desired temperature was reached, the nitrogen pressure was released and the vessel was further purged with hydrogen gas twice, and the hydrogen pressure was maintained at 270 psi.
 - c. The vessel temperature was increased because the hydrogenation reaction was exothermic. After a period of time, the vessel temperature started to decrease and then leveled off until the end of the reaction. (FTIR test has



been a facile method to monitor the reaction progress).

- d. After the reaction was completed, the hydrogen pressure was released slowly, and the purging, followed by releasing of nitrogen gas was repeated twice.

- e. The reaction solution was charged into a flask. A small amount of heptane was used to wash out the remaining catalyst and the wash was combined with the reaction solution in the flask.
 - f. The filtered solution was concentrated in a rotary evaporator to dryness. The residue was further dried *in vacuo*.
 - g. The OH value and degree of hydrogenation were determined using titration and a procedure based on ASTM 1959-97, respectively.
 - h. The viscosities data of the hydrogenated products were determined by a Brookfield DV-III Programmable Rheometer at room temperature.
10. Viscosity of the resultant polymer was determined by me, using a Brookfield DV-III Programmable Rheometer at room temperature, to be 114,000 centipoise at room temperature, and therefore a ratio of viscosity (poise at room temperature) to peak molecular weight raised to the 3.4 power of 0.3×10^{-9} , and therefore within the scope of the claims of the above-referenced reissue application, which claimed an upper limit of 2.0×10^{-9} in some claims, and in 1.0×10^{-9} other claims.
11. I declare under penalty of perjury under the laws of the United States of America that the foregoing is true and correct.


Taejun Yoo

Date: October 10, 2004